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THE REJUVENATION OF A SILICA-"POISONED" ANION EXCHANGER IN URANIUM PROCESSING

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THE REJUVENATION OF A SILICA-"POISONED" ANION
EXCHANGER IN URANIUM PROCESSING

by

A.J. Gilmore

SUMMARY

This investigative study was performed to assess the feasibility and economics of using alkaline reagents, less expensive than costly sodium hydroxide (NaOH), for the rejuvenation of silica-"poisoned" resins. Anion exchange resins are used in the processing of radioactive ores for the separation and recovery of uranium from leach liquors. Silica-"poisoned" resins, arising from the H_2SO_4 leaching of siliceous minerals in the ore, (1) decrease uranium adsorption and desorption efficiencies, and (2), lower exchanger resin capacity for uranium, as illustrated in test results.

An operating mill superintendent can little afford the NaOH rejuvenation more than "once a year", due to the excessive cost of the NaOH reagent. This study concluded that a silica-"poison" removal technique, employing sodium carbonate (Na_2CO_3) rejuvenation plus a NaCl wash, costs only 37% that of the NaOH rejuvenation plus the NaCl wash. Rejuvenation costs, the study indicates, could be further reduced by recycling some of the rejuvenation and NaCl wash solutions. Furthermore, a 13% H_2SO_4 uranium desorption displaced 8.6 times more silica than the uranyl sulphate $[UO_2(SO_4)_3]^{4-}$ adsorption.

LE REJEUNISSEMENT D'UN ÉCHANGEUR D'ANIONS EMPOISONNÉ
DE SILICE DANS LE TRAITEMENT DE L'URANIUM

par

A.J. Gilmore

SOMMAIRE

Une étude a été menée pour évaluer les possibilités et les facteurs économiques concernant l'utilisation des réactifs alcalins, qui sont beaucoup moins chers que l'hydroxide de soude (NaOH), dans le procédé de rajeunissement des résines "empoisonnées" de silice. Les résines d'échange d'anions facilitent la séparation et récupération de l'uranium à partir des liqueurs de lixiviation dans le traitement des minerais radioactifs. Les résultats des essais indiquent que les résines "empoisonnées", qui proviennent de la lixiviation par H_2SO_4 des minéraux silicieux renfermés dans le minerai, ont tendance, en premier lieu, à décroître le rendement de l'adsorption et de la désorption de l'uranium et, en deuxième lieu, réduire la capacité des résines d'échange.

Grâce au prix prohibitif du NaOH , un chef d'un moulin d'exploitation ne peut pas entreprendre le procédé de rajeunissement plus qu'une fois par an. Cette étude a conclu que la technique d'enlèvement du poison de silice qui se sert du carbonate de soude (Na_2CO_3) avec un lavage à NaCl , de 37% moins chère que la méthode de rajeunissement par NaOH plus un lavage à NaCl . L'étude indique qu'on peut réduire davantage le prix de rajeunissement en recyclant une partie des solutions employées dans les procédés de rajeunissement et lavage. En outre, une désorption de l'uranium avec 13% de H_2SO_4 a déplacé en deçà de 8,6 fois plus de silice que l'adsorption avec du sulfate d'uranium [$\text{UO}_2(\text{SO}_4)_3^{4-}$].

INTRODUCTION

The purpose of this work was to examine the feasibility of using alkaline reagents, other than caustic soda (NaOH), for the rejuvenation of silica-"poisoned" anion exchangers. These other reagents would be alternatives to the popular, expensive sodium hydroxide (NaOH). Resin "poisons" affect the economics of the uranium separation process. They decrease the capacity¹ of the exchanger for uranium, and decrease the efficiency² by causing excessive uranium leakage into barren effluents. Silica poisoning appears to be a function of siliceous minerals in radioactive ores, that are solubilized in the H_2SO_4 -leaching process. The chemical change undergone by silica mineral constituents during leaching and solution purification causes silica hydrolysis¹.

The acidity of the uranium liquor, that initially contacts the anion exchanger, is reduced by adsorption of bisulphate (HSO_4^-) anion. This pH increase reduces the stability of silica in the liquor, and the stabilized silica in the liquor can penetrate the matrix of exchangers. The solution inside the resin matrix becomes less acidic than the liquor outside the matrix. Furthermore, the leach liquor in the voids of the resin bed is flushed out with water, at the end of an adsorption phase, with an accompanying pH increase and deposition of silica within resin matrix. Backwashing with water will further increase the formation of precipitated silica by displacing the H^+ -ion from adsorbed HSO_4^- anion on resin resulting in a further pH increase within resin bead. These conditions of silica hydrolysis result in coagulation and precipitation of silica within the resin matrix. This hydrolyzed silica interference, with the rate-controlling diffusion of anions in and out of resin bead, results in poor adsorption characteristics², and the resin bead may rupture through failure of resin cross-linkage. Figure 1 is a diagrammatic representation of the structure of a resin bead. The straight lines indicate polystyrene chains cross-linked to one another. The N-represents an active anion exchange group. Silica "poisons", once adsorbed, will not desorb readily with conventional desorbing reagents used in the uranium desorption phase.

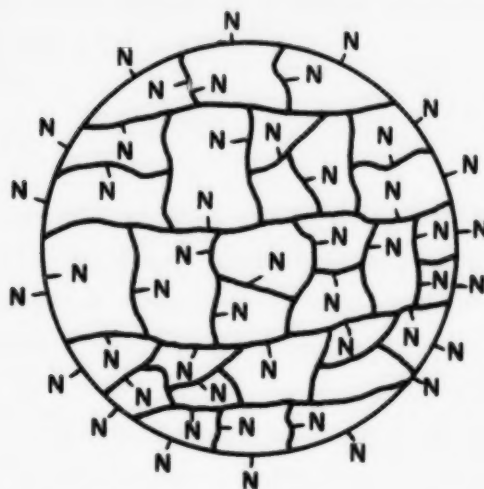


FIGURE 1. A DIAGRAMMATIC REPRESENTATION OF THE STRUCTURE OF AN ANION-EXCHANGE RESIN BEAD (2).

Rejuvenation of a silica "poisoned" anion exchanger is normally accomplished with expensive caustic soda (NaOH) at 27.1¢/lb⁽³⁾ or 58.8¢/kg. NaOH has become a noticeable operating expenditure. Mr. W. Davies, marketing manager of Rohm and Haas, Toronto, at a 1980 CUPMC, suggested that Madawaska Mines Ltd. perform a "caustic rejuvenation once a month" instead of "once a year" as had been the practise. The Madawaska mill super-intendent, P. Kresin replied, "Madawaska can't afford a NaOH rejuvenation once a month!" Any costly reagents, labor and downtime used to remove the silica "poison", and rejuvenate the anion exchanger, are also an accountable operating cost. Milder reagents such as sodium carbonate (Na₂CO₃) at 4.2¢/lb⁽³⁾ or 9.26¢/kg or potassium carbonate (K₂CO₃) at 11.75¢/lb⁽³⁾ or 25.9¢/kg may reduce the "osmotic shock" to which resin is subjected in rapid changes from one resin form to another with when using relatively strong reagents such as NaOH and H₂SO₄, i.e. SO₄-form $\xrightarrow{\text{NaOH}}$ OH⁻-form $\xrightarrow{\text{H}_2\text{SO}_4}$ HSO₄⁻-form. Rupture of the resin may occur with such severe changes or shocks to the resin bead.

Sodium carbonate (Na_2CO_3) and K_2CO_3 reagents were examined in this testwork as alkaline rejuvenation reagents to cleanse an anion exchanger deliberately "poisoned" with the silica (SiO_2).

PROCEDURE

A rejuvenation process was chosen, for the silica-"poisoned" exchanger, to encompass all the reviewed¹ industrial rejuvenation processes in practise. The technical methods chosen to investigate the capabilities of three alkalies to rejuvenate a silica-"poisoned" anion exchanger were as follows:

1. An anion exchanger was loaded with uranium in a synthetic uranyl sulphate liquor, followed by a displacement water wash.
2. The adsorbed uranium was desorbed with 12% H_2SO_4 , plus a displacement water wash.
3. The resin was converted from the $\text{SO}_4^{-2}/\text{HSO}_4^{-}$ -form to the $-\text{OH}$ form⁴ by:
 - (a) an initial backwash,
 - (b) introduction of 4 lb NaOH /cu ft resin, as 4% NaOH at desorption flowrate (i.e. 10 minutes retention time),
 - (c) water displacement wash at desorption rate,
 - (d) backwash to classify bed and get rid of air bubbles,
 - (e) repeat (b),
 - (f) repeat (c),
 - (g) fast rinse to rid resin of NaOH at adsorption flowrate (i.e. 2 minutes retention time). The OH -form resin was expanded by 28%.
4. The $-\text{OH}$ form of 400 mL of Amberlite IRA-400 was contacted with a pH 1.92 silica solution containing 1.04 g Si/litre at a downflow rate of 50 mL/min. Resin/solution contact was continued until effluent Si-concentration was equivalent to influent concentration. Resin was backwashed to classify and mix the resin bed, with 2 bed volumes of water, and then allowed to settle.
5. The Si-form resin was removed from the column and vacuum-treated on a buchner filter to remove bulk of moisture. The partially air-dried resin was divided into 4 equal parts of 87 g (=100 mL) apiece.

6. Contact each of 3 parts of resin with 300 mL (3 bed volumes) of each alkali (5% NaOH, 10% Na₂CO₃ and 10% K₂CO₃) by air agitation, in 1"-ID Pyrex glass column (Figure 2), for 7 to 8 hours.
7. Drain alkali from column, after each 7-8 h period, then replace with 300 mL of fresh similar alkali and air agitate as in (6).
8. Repeat (7).
9. Drain spent alkali for analysis then wash with 10% NaCl until effluent pH was 7 or 12-14 bed volumes. This is to minimize resin shock and displace Si from resin. This was followed by a displacement wash.

RESULTS

ADSORPTION AND DESORPTION OF AMBERLITE IRA-400

The uranium influent solution was prepared from a stock reagent of 99.8% UO₃, MgSO₄·7H₂O and 1-1 H₂SO₄ to give a uranyl-sulphate liquor containing:

0.75 - 0.85 g U/litre
39.6 - 41.7 g SO₄/litre
and pH 1.5

This liquor was fed to 400 mL of the anion exchanger, in a Pyrex brand glass pipe 1 in. ID x 5 ft or 2.54 cm ID x 1.52 m length at the rate of 0.125 bed volumes/min. The adsorption curve for loading uranium on Cl-form of fresh resin bed is presented in Figure 3.

The loaded uranium column was desorbed with 12% H₂SO₄, after a displacement wash, at a flowrate of 0.04 bed volumes/min for 12 bed volumes (Figure 4). The resin loading was equivalent to 62.76 g U/litre resin or 3.92 lb U/cu ft or 62.7 kg/m³ resin. The resin bed was water washed to displace the 12% H₂SO₄ from resin bed.

ADSORPTION OF SILICA (Si) ON AMBERLITE IRA-400

The influent silica solution was prepared from an alkaline technical grade Na-silicate (40-42 °Be) solution adjusted to pH 1.92 with 1-1 H₂SO₄ to assay 1.04 g Si/litre solution. This acidified solution was introduced to the OH-form resin column of Amberlite IRA-400 at 0.125 bed volumes/min

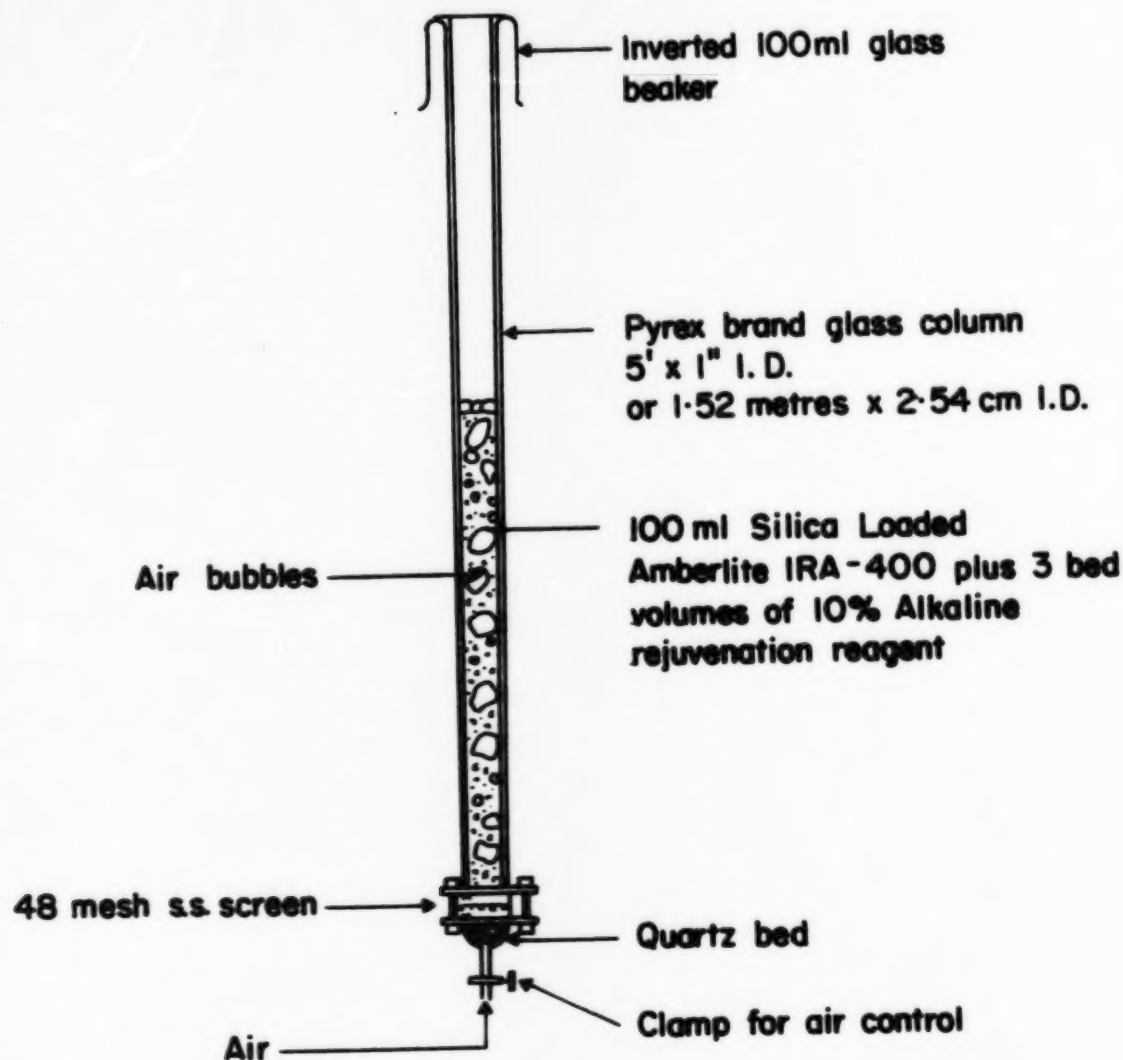


FIGURE 2. SET-UP FOR NaOH , Na_2CO_3 AND K_2CO_3 RESIN REJUVENATION.

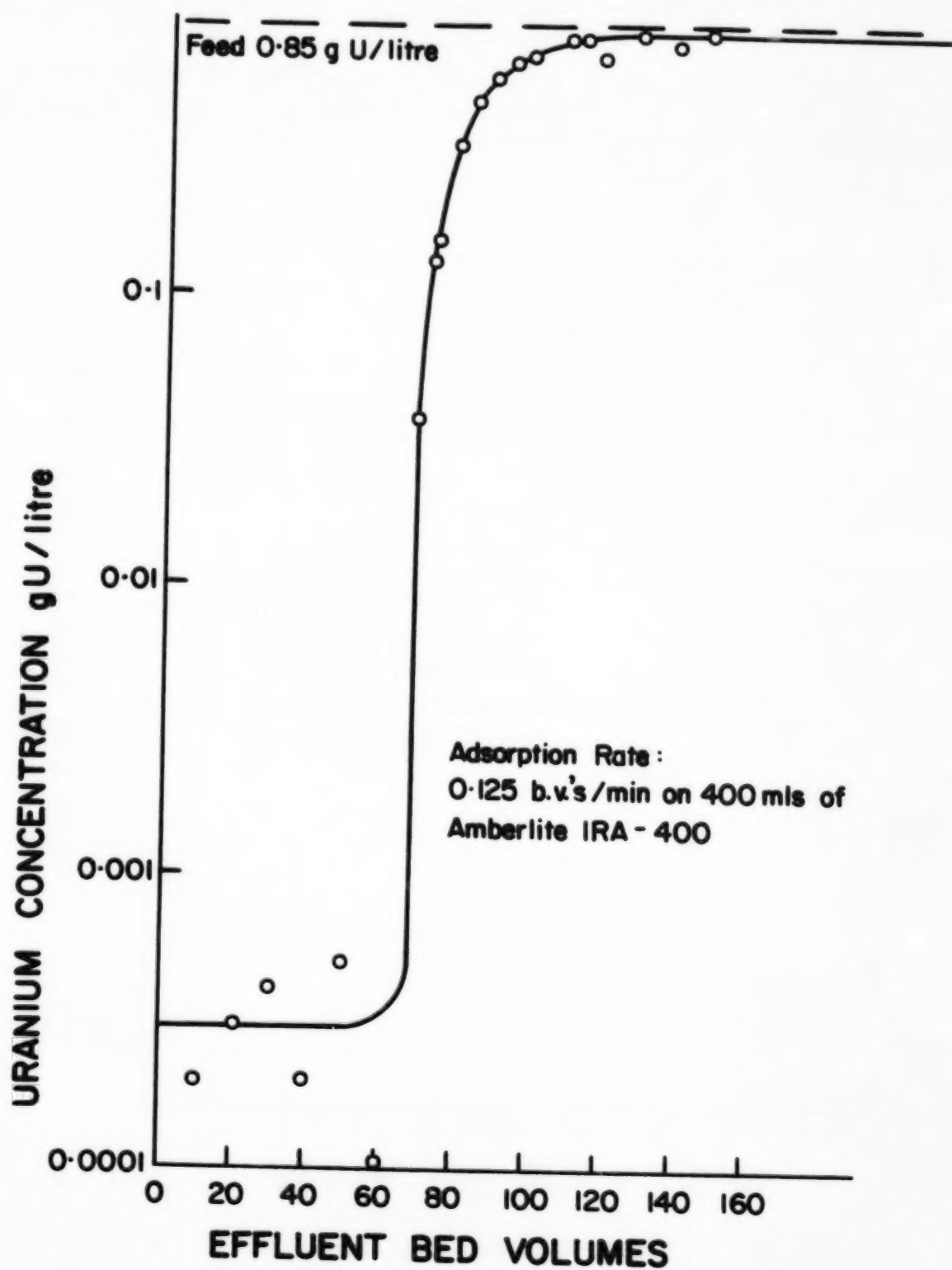


FIGURE 3. AN ADSORPTION OR LOADING CURVE OF URANIUM ON AMBERLITE IRA-400 RESIN.

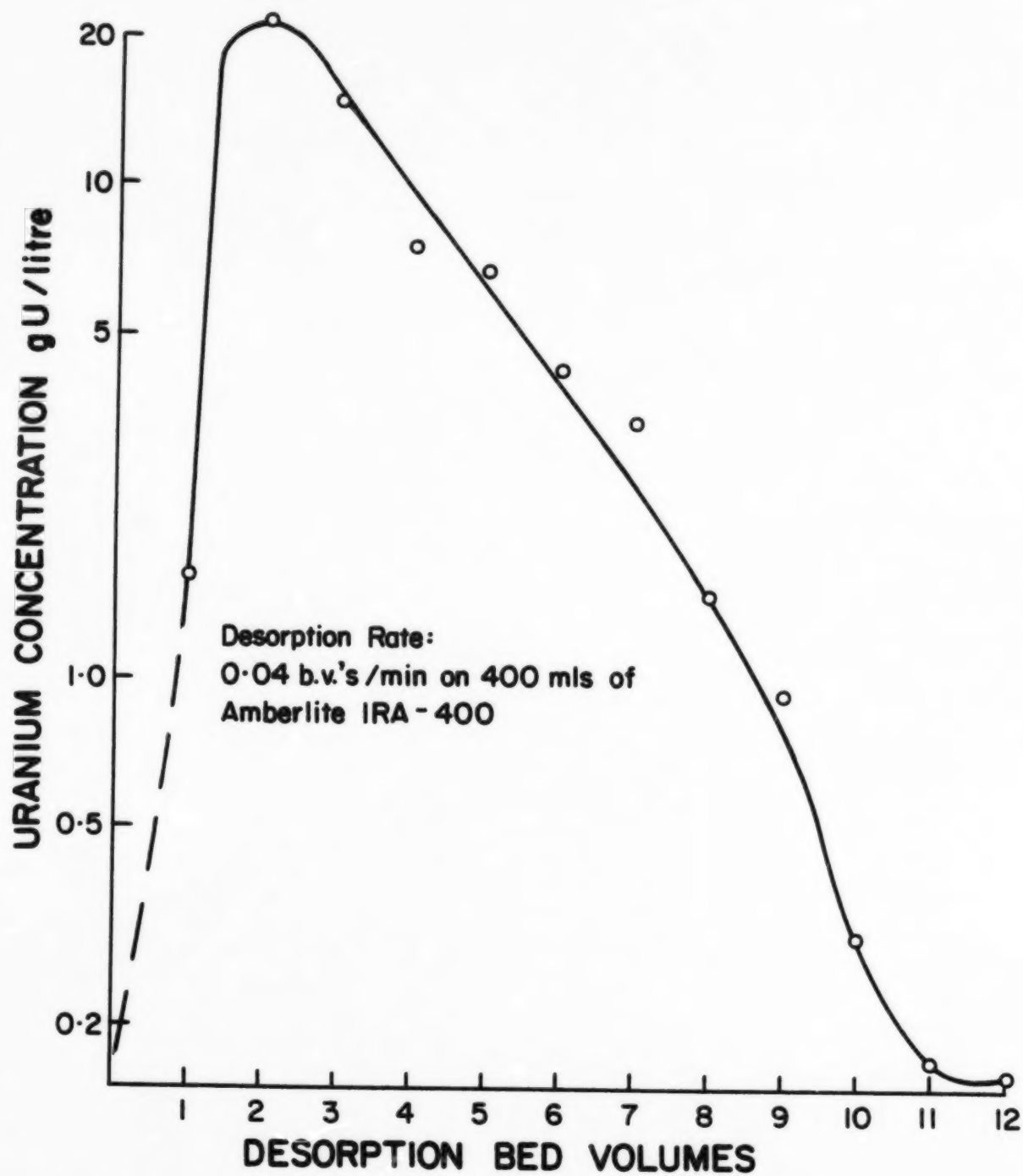


FIGURE 4. DESORPTION OF URANIUM FROM AMBERLITE IRA-400 WITH 12% H_2SO_4 .

until the Si concentration of effluents = Si concentration of influent. The Si-loading, from analyses of effluent samples, was 18.15 g Si/litre resin or 1.13 lb Si/cu ft or 18.08 kg Si/m³ IRA-400 under test conditions. The silica loading curve is shown in Figure 5. The decreasing pH in Figure 5, illustrates the change of resin form from alkaline to acidic form with eventual Si-loading. An upflow water wash was applied to the resin bed of IRA-400 to mix and classify the resin bed.

The procedure for dividing Si-"poisoned" resin bed into 4 parts has already been described.

REJUVENATION OF Si-"POISONED" IRA-400 WITH ALKALIS

(a) Rejuvenation with Sodium Hydroxide (NaOH)

A rejuvenating solution, 300 mL of 5% NaOH (48.7 g NaOH/litre) was contacted with "poisoned" resin in a 5 ft x 1 in. ID or 1.52 m x 2.54 cm glass column, as in Figure 2, and agitated with an upflow addition of air for 7-8 hours. Then the alkali solution was drained out the bottom of column. The resin was held in column on a quartz bed and a 48-mesh stainless steel screen. The same resin bed was similarly treated twice more. The alkali consumed after the third 5% NaOH contact was 4.32 g NaOH. A conception of the chemistry is:



The NaOH rejuvenated resin was transferred to a 100 ml burette column for a 10% NaCl wash. Silica removed was 1.469 g Si

(b) Rejuvenation with Sodium Carbonate (Na₂CO₃)

A second portion of Si-"poisoned" resin was contacted 3 times with 300 mL fractions of 10% Na₂CO₃ (103 g Na₂CO₃/litre) and air-agitated as in (a) above. The alkali consumed after the third 10% Na₂CO₃ contact was 3.77 g Na₂CO₃. A solution of 10% Na₂CO₃ becomes caustic with a pH of 11.3 as:



The NaOH then attacks the silica as in equation (1). The second portion of resin was transferred to a 100 mL burette for a 10% NaCl wash, after being contacted 3 times with 10% Na₂CO₃. Silica removed was 0.8096 g Si.

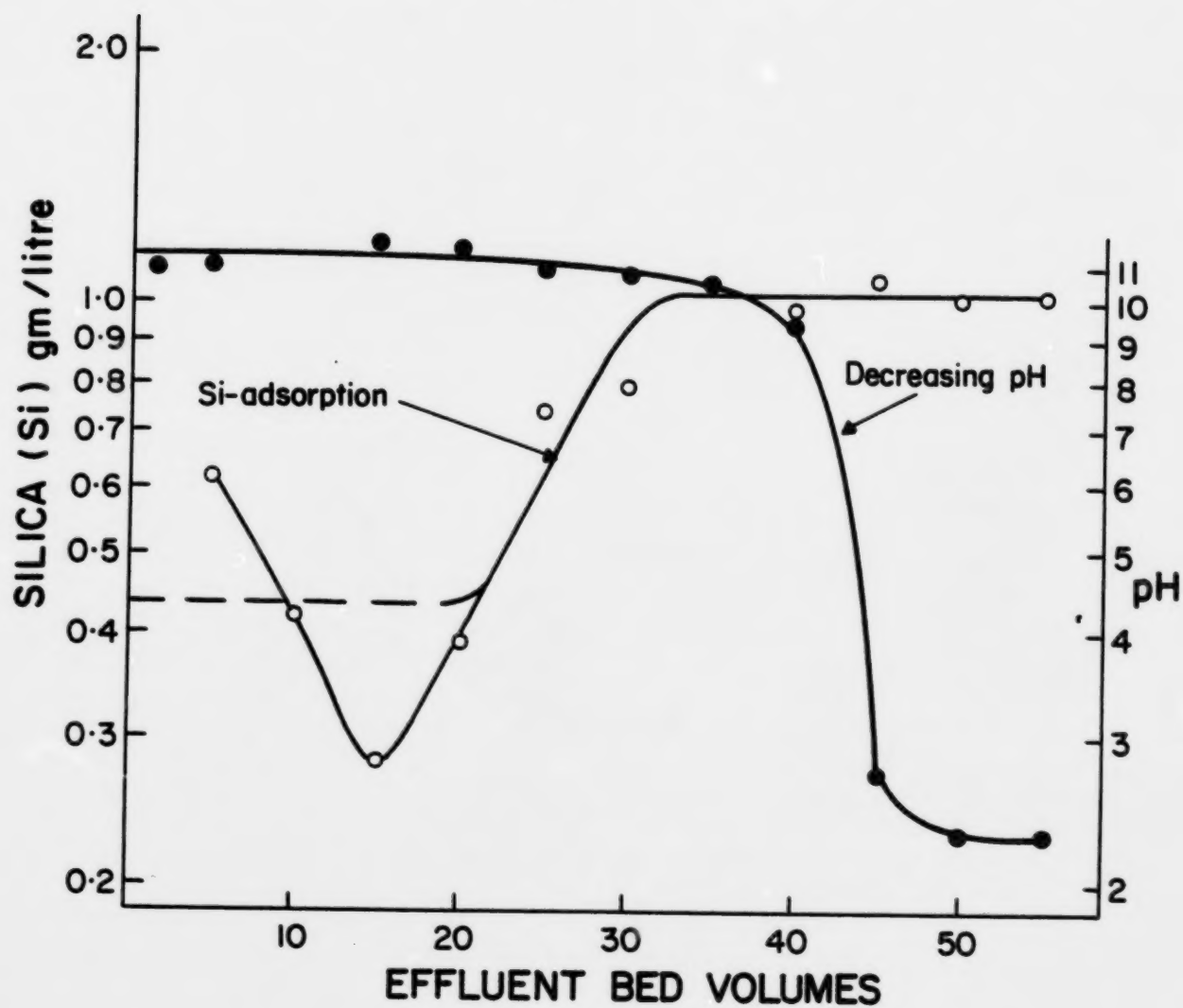


FIGURE 5. THE ADSORPTION LOADING OF SILICA (Si) ON OH-FORM AMBERLITE IRA-400 RESIN.

(c) Rejuvenation with Potassium Carbonate (K_2CO_3)

A third portion of Si-"poisoned" resin was similarly contacted 3 times in column with 300 mL portions of 10% K_2CO_3 (98.8 g K_2CO_3 /L) then air-agitated as in (a) above. The total alkali consumed after third 10% K_2CO_3 contact, was 12.87 g K_2CO_3 . The 10% K_2CO_3 became caustic at pH of 11.6 as:



The silica in resin is then attacked by the KOH as:



This third portion of resin, after third contact with 10% K_2CO_3 , was transferred to a 100 mL burette for a 10% NaCl wash. Silica removal was 0.6678 g.

10% NaCl Wash of Alkali Rejuvenated Resin

Each of the NaOH, Na_2CO_3 and K_2CO_3 rejuvenated resins was individually washed with 10% NaCl (106 g NaCl/litre) in a 100 mL burette at 0.02 bed volumes/min, (1) to minimize "osmotic" shock or resin rupture due to making rapid pH changes from one resin form to another with strong reagents such as the alkalis and acids, (2) to displace any silica removable with the Cl-anion. Figure 6 illustrates the silica desorbed from each of the alkali rejuvenated resin portions. The Na_2CO_3 and K_2CO_3 curves imply that there is still silica on the second and third resin portions. The NaCl, in each of the 100-mL rejuvenation columns, was displaced with water.

Silica Removed by Alkalis plus NaCl

Table 1 shows the silica removed by the alkalis regeneration plus NaCl displacements.

Silica Displaced by U-Adsorption and H_2SO_4 Desorption

Figures 7 and 8 illustrate the Si displaced and desorbed during an anion-exchange uranium recovery cycle on the 4th portion of Si-"poisoned" resin. Figure 7 shows the silica 'bumped off' or displaced from the resin

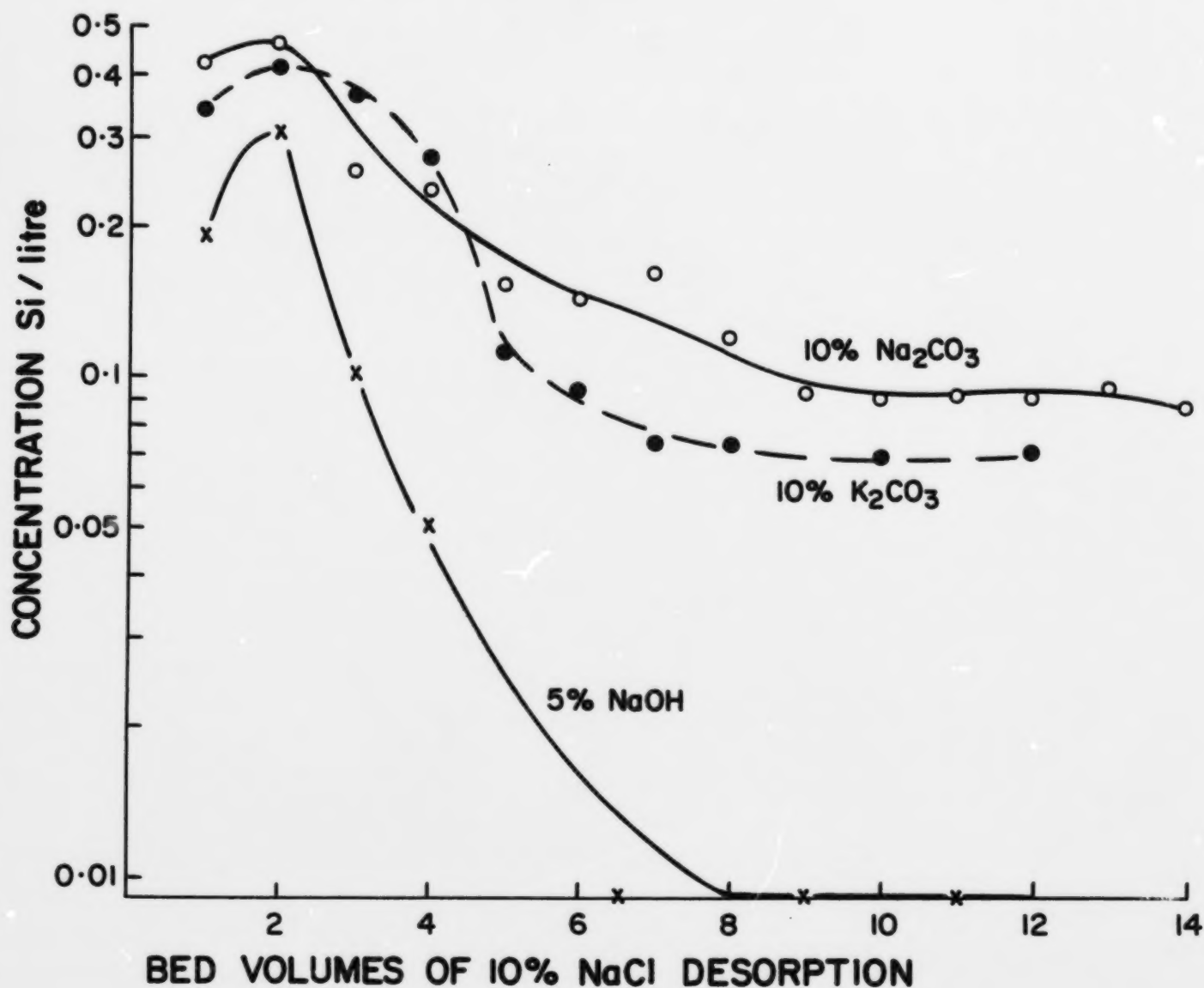


FIGURE 6. SILICA DESORPTION, WITH 10% NaCl, OF VOLUMES OF AMBERLITE IRA-400 FORMERLY REJUVENATED WITH 5% NaOH, 10% Na₂CO₃ AND 10% K₂CO₃ FOR SILICA REMOVAL FROM THE EXCHANGER.

Table 1 - Silica removal from "poisoned" resin by alkali regeneration and 10% NaCl wash

Stage	5% NaOH					10% Na ₂ CO ₃					10% K ₂ CO ₃				
	Sample Vol. mL	Silica		NaOH		Sample Vol. mL	Silica		Na ₂ CO ₃		Sample Vol. mL	Silica		K ₂ CO ₃	
		Si g/litre	g Si Removed	NaOH g/litre	g NaOH Consumed		Si g/litre	g Si Removed	Na ₂ CO ₃ g/litre	g Na ₂ CO ₃ Consumed		Si g/litre	g Si Removed	K ₂ CO ₃ g/litre	g K ₂ CO ₃ Consumed
1	320	3.44	1.101	46.1	0.832	370	0.48	0.178	83.4	0.042	280	0.41	0.115	71.9	9.508
2	295	0.77	0.227	43.4	1.564	290	0.64	0.186	96.8	2.828	295	0.56	0.165	91.2	2.736
3	320	0.21	0.067	42.7	1.920	300	0.68	0.204	100.0	0.900	300	0.65	0.195	96.7	0.630
Total Alkali					4.315 g					3.77 g					12.87
10% NaCl Wash			0.074					0.242					0.193		
g NaCl Consumed					g NaCl 20.06					g NaCl 18.41					g NaCl 18.26
Total g Si Removed			1.469					0.810					0.668		

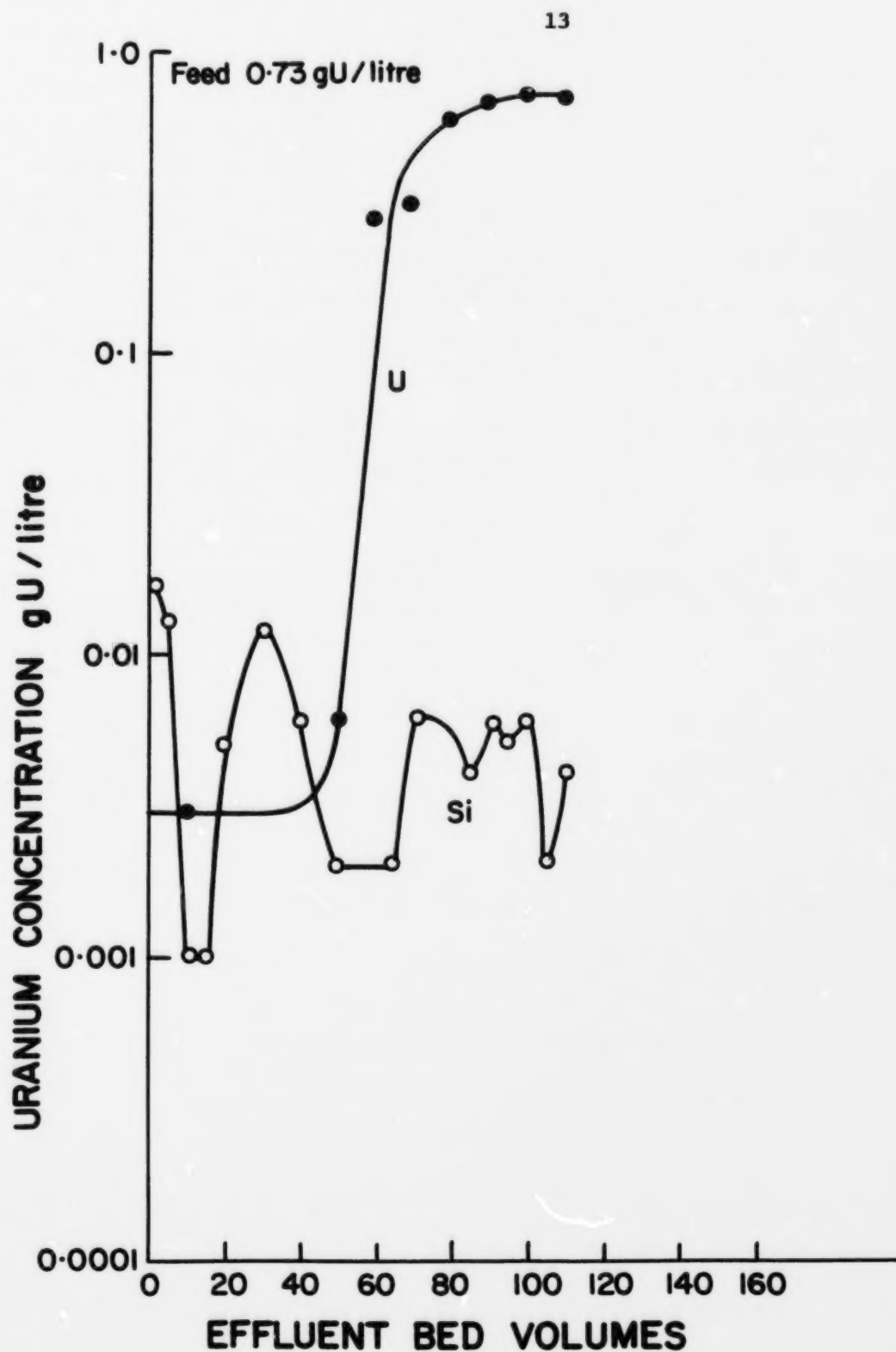


FIGURE 7. THE ADSORPTION OF URANIUM ON SILICA-POISONED AMBERLITE IRA-400.

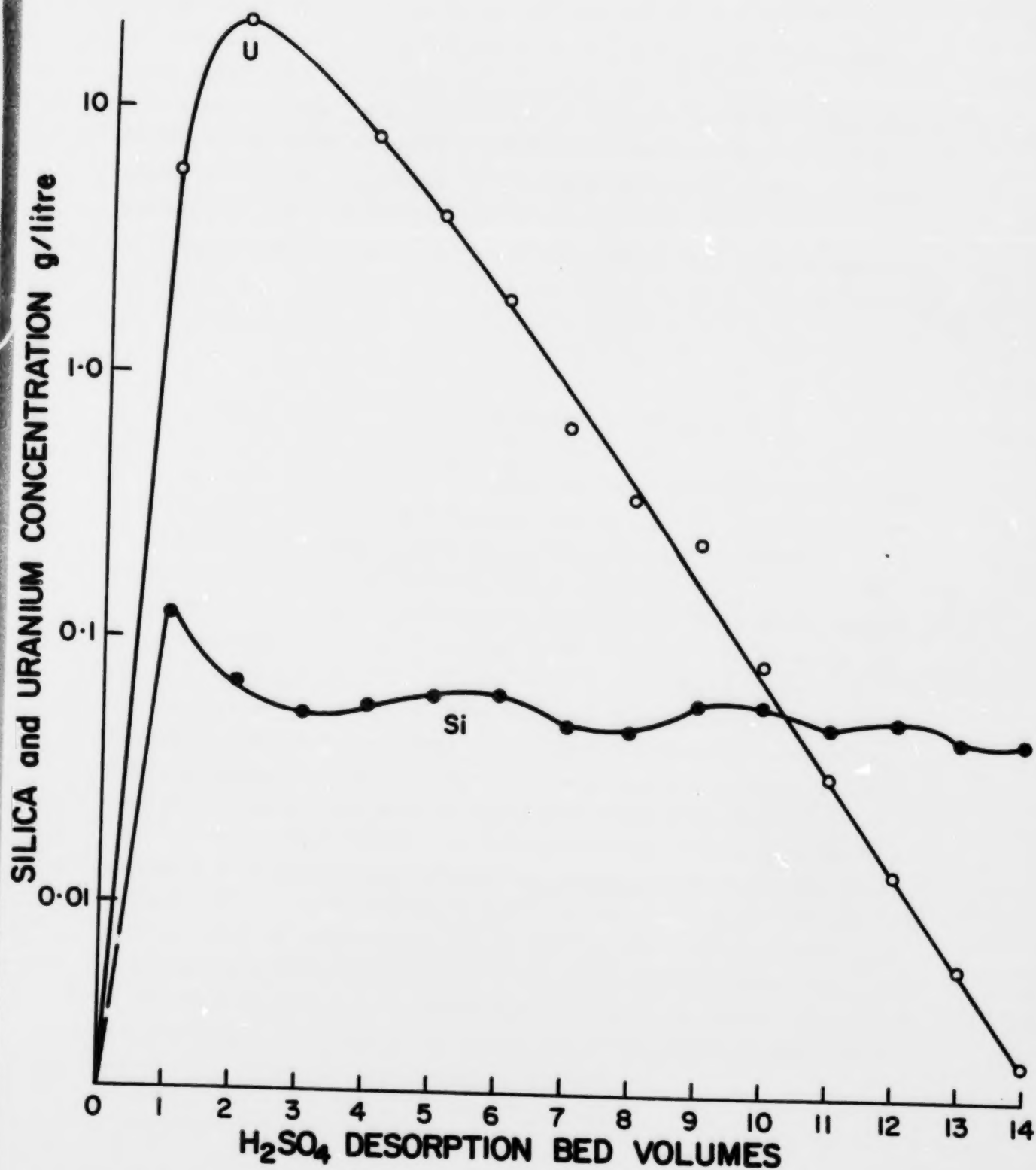


FIGURE 8. THE DESORPTION OF URANIUM AND SILICA FROM A SILICA-POISONED AMBERLITE IRA-400 WITH 13% H_2SO_4 .

during the adsorption of the uranyl-sulphate complex $[\text{UO}_2(\text{SO}_4)_3]^{4-}$. The Si 'bumped off' is 0.098 g Si/litre resin and decreases as U-adsorption progresses.

Figure 8 shows the Si displaced from the anion exchanger, along with the $\text{UO}_2(\text{SO}_4)_3^{4-}$ desorbed during a 13% H_2SO_4 desorption. The silica displaced during H_2SO_4 desorption is 0.840 g Si/litre resin and is 8.6 times greater than the Si displaced with $\text{UO}_2(\text{SO}_4)_3^{4-}$.

The uranium loading is 55.63 g U/litre resin, which is an U-capacity loss of 11.4%, compared to initial capacity loading of 62.76 g U/litre resin.

DISCUSSION

"Resin shock" is reduced by converting resin from OH-form to Cl-form, with NaCl, before 'on service' contact with strong acid in uranium process liquors. This treatment also minimizes the formation of hydroxy precipitates of U, Fe, Si and etc.

The silica content, in our synthetic liquor, was 1040 ppm Si whereas the silica content of Madawaska pregnant liquors to ion exchange was 257 ppm Si i.e. only about 24% of Si-content of the synthetic liquor.

The adsorption of silica, from Madawaska pregnant liquors containing 257 ppm Si, "by the ion-exchange resin is serious enough that after 9-12 months of operation, the resin adsorption and elution kinetics are lost. Caustic rejuvenation was done annually since start-up in 1976"⁵.

A further communication from Madawaska⁶, after this program was underway, presented silica contamination loadings on Amberlite IRA-400 to be 0.234 to 0.421 lb Si/cu ft or 3.74 to 6.74 kg Si/m³ resin. This is only 20.7% to 37.3% of synthetically loaded IRA-400 with Si, but is sufficient to cause a noticeable loss of kinetics on adsorption and desorption. Then, it is unnecessary to treat a silica saturated anion exchanger in this study when treating about a 40% silica-loaded resin would be sufficient - or a commercially "poisoned" resin would be preferable.

The silica removed with 10% Na_2CO_3 rejuvenation + NaCl desorption removed 0.810 g Si, which is equivalent to 0.506 lb Si/cu ft or 120% of Madawaska "poisoned" resin. The 10% K_2CO_3 rejuvenation + NaCl desorption removed 0.668 g Si, equivalent to 0.417 lb Si/cu ft. So the Na_2CO_3 + NaCl treatment is more than adequate to treat the Madawaska resin, while the K_2CO_3 + NaCl treatment is border line.

Table 2 presents an estimated economic comparison of rejuvenation alkalis + NaCl washes for removal of silica from Si-poisoned resins.

Table 2 - Estimated economic comparison of rejuvenation alkalis and NaCl wash of Si-"poisoned" resins

Operation	NaOH @ 27.1¢/lb ³ or 59.8¢/kg	Na_2CO_3 @ 4.2¢/lb ³ or 9.3¢/kg	K_2CO_3 @ 11.75¢/lb ³ or 25.9¢/kg
Alkali Rejuvenation	\$ 0.73/cu ft or \$ 0.021/m ³	\$0.099/cu ft or \$0.0028/m ³	\$0.944/cu ft or \$0.027/m ³
10% NaCl Wash (NaCl @ 0.025¢/lb ⁽³⁾) (or 0.055¢/kg)	0.31/cu ft or 0.0088/m ³	0.287/cu ft or 0.0081/m ³	0.285/cu ft or 0.0081/m ³
Total Cost Alkali + NaCl	\$ 1.04/cu ft or \$ 0.029/m ³	\$0.386/cu ft or \$0.0109/m ³	\$1.23/cu ft or \$0.035/m ³
Reagent Cost /425* cu ft resin or /12.03 m ³	<u>\$442.00</u>	<u>\$164.05</u>	<u>\$522.49</u>
Reagent Cost /lb U or /kg U	<u>\$0.265</u> <u>\$0.584</u>	<u>\$0.098</u> <u>\$0.216</u>	<u>\$0.314</u> <u>\$0.692</u>

*Each Madawaska column holds 425 cu ft or 12.03 m³ of anion-exchanger resin⁵.

The results show the 10% Na_2CO_3 + 10% NaCl rejuvenation treatment procedure costs 37% of the 5% NaOH + 10% NaCl rejuvenation technique.

The sodium carbonate rejuvenation of anion-exchanger resins for Si-poisoning would be advantageous (1) economically, and (2), a less harsh treatment than the NaOH technique. The carbonate treatment would not be expected to degrade the amine resin to cause the strong "fish" or amine odor evolved with the NaOH-rejuvenation - which overtreats the resin in Si-removal.

A saving in alkali rejuvenation reagent would result if the alkaline Si-leach solution, from the first rejuvenation contact, was discarded, and the leach solutions from the second and third rejuvenation contacts were stored for recycle to a subsequent rejuvenation on a fresh batch of "poisoned" resin. A fresh batch of alkali rejuvenation solution would be prepared for third and last contact. Similarly, the first two bed volumes of NaCl washes could also be discarded. The balance of the NaCl bed volumes could be stored for recycle to subsequent NaCl washes. Two bed volumes of fresh 10% NaCl would be introduced at end of NaCl wash cycle.

It might be advantageous, inexpensive and easy to treat about 1/10 of resin capacity continuously with Na_2CO_3 to combat Si-poisoning of resin. Ion-exchange plants with built-in rejuvenation circuits¹, have been less prone to Si-poisoning.

Figures 6 and 7, showing Si displaced with uranium adsorption and an H_2SO_4 desorbing solution are the first indication, of which this writer is aware, of the manner in which Si is displaced from a silica-poisoned anion exchanger.

CONCLUSION

An investigation has demonstrated that a Na_2CO_3 rejuvenation plus a NaCl wash, of a commercially silica-poisoned anion exchanger, is capable of silica removal with only 37% of the reagent costs required for a NaOH rejuvenation plus NaCl wash. This technique should be attempted on the next available commercial resin with silica "poisoning" that produces loss of kinetics on adsorption and desorption.

The recycling of rejuvenation solutions and NaCl washes, as discussed but not tried experimentally, would effectively lower rejuvenation reagent costs still further.

The recycling of rejuvenation solutions and NaCl washes, as discussed but not tried experimentally, would effectively lower rejuvenation reagent costs still further.

In the silica displacement on adsorption and desorption, (1) the amount of silica 'bumped off' decreases as the $\text{UO}_2(\text{SO}_4)_3^{4-}$ adsorption progresses (Figure 6), and (2), the amount of silica displaced decreases slowly as H_2SO_4 -desorption progresses (Figure 7). The 13% H_2SO_4 desorption

displaced = 8.6 times as much silica as the $\text{UO}_2(\text{SO}_4)_3^{4-}$ adsorption. The resin capacity of uranium decreased by 11.4% on silica "poisoned" resin.

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